

**A STUDY ON EXTRACTION OF *ZINGIBER OFFICINALE* OIL BY USING
SOXHLET DISTILLATION**

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ABSTRACT

Ginger is the common name for the *Zingiber officinale* which is one of the plants that belongs to the *Zingiberaceae* family. Ginger is a very useful herb plant that is usually indigenous to the Jamaica, India, China, Indonesia, Nigeria and Southeast Asia. It is found that zingiberene is the main compound in the ginger. The ginger oil is widely used in both traditional and contemporary natural medicine and also is commercialized for use in food as well. The objective of this research is to extract ginger oil by using Soxhlet Distillation, to evaluate the effect of different solvent on the extraction and to investigate the effect of extraction time. Four types of solvents are used in this research which is methanol, dichloromethane, benzene and acetone while the extraction time is varied about 4 hours, 6 hours, 8 hours and 10 hours. After extracting the oil using Soxhlet Distillation, the oil then is separated from the solvent by rotary evaporator and finally the sample was analyzed using GC-MS in order to identify the components in the oil. Methanol gave highest yield of the *Zingiber Officinale* oil at 8 hours extraction time which is 27.33 % of mass yield.

ABSTRAK

Halia adalah nama umum untuk *Zingiber officinale* yang merupakan salah satu tanaman yang tergolong dalam keluarga *Zingiberaceae*. Halia merupakan tanaman herba yang sangat berguna yang biasanya berasal dari Asia, Jamaica, India, China, Indonesia, Nigeria dan Tenggara. zingiberene adalah sebatian utama dalam halia. Minyak halia diguna meluas dalam kedua-dua ubat tradisional dan kontemporari dan juga dikomersialkan untuk digunakan dalam makanan juga. Tujuan kajian ini adalah untuk mengekstrak minyak halia dengan menggunakan Soxhlet Penyulingan, untuk menilai pengaruh pelarut yang berbeza pada ekstraksi dan untuk menyiasat pengaruh waktu ekstraksi. Empat jenis pelarut yang digunakan dalam kajian ini adalah metanol, dichloromethane, benzena dan aseton sementara waktu ekstraksi adalah 4 jam, 6 jam, 8 jam dan 10 jam. Setelah mengekstrak minyak menggunakan Soxhlet Penyulingan, minyak kemudian dipisahkan dari pelarut dengan rotary evaporator dan akhirnya sampel dianalisis menggunakan GC-MS untuk mengenal pasti bahagian-bahagian dalam minyak. Metanol memberikan hasil tertinggi minyak halia pada waktu ekstraksi 8 jam iaitu sebanyak 27.33 % jisim hasil.

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LIST OF ABBREVIATIONS

ASE	Accelerated Solvent Extraction
PEE	Petroleum Ether Extracts
UAE	Ultrasound Assisted Extraction
MAE	Microwave Assisted Extraction
GC-MS	Gas Chromatography- Mass Spectrometer
FMASE	Focused Microwave-Assisted Soxhlet Extraction

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Essential oils are one of the demanded natural products in the local or international market. Essential oils are extracted from many different parts of their plants. Essential oils do not as a group have any specific chemical or pharmaceutical properties in common. Instead they are defined by the fact that they convey characteristic fragrances. It follows that the common tendency to speak of essential oils as a category, as if that implied anything in particular about their medical, pharmacological, or culinary properties, is highly unreliable and often actually dangerous.

There is various extraction methods are used in the manufacture and extraction of essential oils and the method used is normally dependant on what type of material is being used. So one of the method is a distillation. This method converts the volatile liquid (the essential oils) into a vapor and then condenses the vapor back into a liquid. Distillation is a method of separating mixtures based on differences in their volatilities in a boiling liquid mixture. Distillation is a unit operation, or a physical separation process, and not a chemical reaction. It is the most popular and effective method in use today in producing essential oils.

1.2 Problem Statement

Until recently, ginger is a very useful herb plant which is widely used in both traditional and contemporary natural medicine. In order to fulfill the demand of ginger oil in this country, the research about this product should be carried out to find the best technique and the cheapest way to obtain the higher yield of the product.

Soxhlet Distillation is one of the conventional methods used in extraction of essential oils but there is lack of documentation or research publications on this part that are contributed. So, this research is actually to prove that the method can be used to produce essential oils and at the end will give some information on extraction condition in order to produce highest production. Soxhlet distillation is used in order to extract the ginger oil due to its efficiency to produce high yield of essential oil. This is because there is certain method that cannot extract some components but by using the Soxhlet Extarctor, they could be extracted well.

There are many factors that can give an effect in the extraction process. One of the factors is solvent extraction. Solvent extraction is usually used to recover a component from either a solid or liquid. The sample is contacted with a solvent that will dissolve the solutes of interest. So it is related to the solubility and viscosity of the solvent itself in order to extract the materials.

The other factor is extraction time. Extraction time has its own effect on the extraction where increasing of extraction time will increase the extracts of the oil. This research is purposes to determine the best extraction time to produce most yield of oils.

1.3 Objectives

The objectives for this research are as follows:

- i. To extract ginger (*Zingiber Officinale*) oil by using Soxhlet Distillation.
- ii. To study the effect of different solvent used on the extraction of ginger oil.
- iii. To study the effect of extraction time on the extraction of ginger oil.

1.4 Scopes of Research

In order to achieve the objectives that have been prescribed before, this research is focused on two main scopes which are:

- i. Ginger or *Zingiber Officinale* is used as a sample.
- ii. Investigate the effect of solvent used in the extraction of ginger oil by using methanol, dichloromethane, benzene and acetone as a solvent.
- iii. Investigate the effect of extraction time in the extraction of ginger oil by varying the time for 4 hours, 6 hours, 8 hours and 10 hours.
- iv. The chemical components in the extracted oil were determined by analysis of Gas Chromatography-Mass Spectrometer (GC-MS).

1.5 Rationale and Significant

This research is study in order to obtain the optimal or the best condition for the extraction of the *Zingiber Officinale* oil by using Soxhlet Distillation.

CHAPTER 2

LITERATURE REVIEW

2.1 Ginger (*Zingiber Officinale Roscoe*)

Ginger is a very useful herb plant that originated from India, China and Java and yet is also native to Africa and the West Indies. It is indigenous to the Asia Southeast (Khaki *et.al.*, 2009; K. C. Zancan *et.al.*, 2002; Khairu Aizam, 2006; Lee, 2007) especially in Indo-Malaysia. It is stated that the main producer of ginger is in Jamaica (Alfaro *et.al.*, 2003; Amir Shadmani, 2004; Khairu Aizam, 2006) and also with India, China, Indonesia and Nigeria (Wohlmuth *et.al.*, 2006).



Figure 2.1: *Zingiber Officinale* Rhizome (adapted from wordpress.com)



Figure 2.2 : Plant of *Zingiber Officinale*

Ginger is scientifically name as *Zingiber Officinale Roscoe* which is one of the plants that belongs to the *Zingiberaceae* family and it is a monocotyledonous (Jiang *et.al.*, 2005; K. C. Zancan *et.al.*, 2002; Lee, 2007; Natta *et.al.*, 2008).

Two major groups of compounds which is including gingerol related compounds and diarylheptanoids have been reported as a bioactive components from this plant. 2,3 Gingerol related compounds, comprising distinct groups which is homologous series that are differentiated by the length of their unbranched alkyl chains is actually have recently gained attention in a variety of biological activity studies (Jiang *et.al.*, 2005) and it is found that zingiberene is a major component of a *Zingiber Officinale* (K. C. Zancan *et.al.*, 2002; Yang *et.al.*, 2009) and its derivatives, and compounds of pharmacological activity of ginger are gingerols and its derivatives.

2.1.1 Main Chemical Compound in *Zingiber Officinale* (Zingiberene)

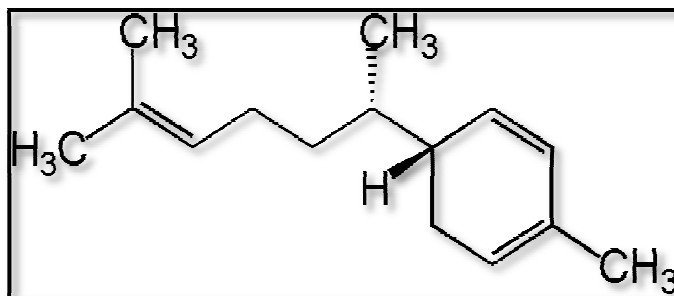


Figure 2.3 : Molecular Structure of Zingiberene
(adapted from wikimedia.org, 2010)

Zingiberene is a monocyclic sesquiterpene that is the predominant constituent of the oil of *Zingiber officinale* from which it gets its name. It is synonyms as a (S-(R*,S*))-(5-(1,5-dimethyl hexen-4-yl)-2-methyl-1,3-cyclohexa-1,3-diene or (S-(R,S))-(5-(1,5- dimethyl-4-hexenyl)-2-methyl-1,3-cyclohexadiene or (5R)-2-methyl-5-[(2S)-6-methylhept-5-en-2-yl]cyclohexa-1,3-diene or α -zingiberene.

2.1.2 Uses of *Zingiber Officinale*

Ginger also known as *Zingiber Officinale* is widely used in both traditional and contemporary natural medicine (Haghighi *et.al.*, 2005; Jiang *et.al.*, 2005; K. C. Zancan *et.al.*, 2002; Yang *et.al.*, 2009). It is stated that the ginger has been used in medicinally in India since ancient times (Lee *et.al.*, 2007)

In Chinese and Unani-Tibb systems of medicine, ginger is used to treat catarrh, rheumatism, nervous diseases, gingivitis, toothache, painful menstruation, asthma, stroke, constipation, diabetes and migraine. In Asian medicine, it is used as a carminative which is known as digestive aid, stimulant, diuretic and anti-emetic. Patients with chronic painful disease often seek alternative therapy and currently ginger is one of the most popular herbal medications for rheumatic diseases. Ginger

(*Zingiber officinale*) has been used for medicinal purposes since antiquity. In particular, it has been an important plant for the traditional Chinese and Indian medicines (Haghighi *et.al.*,2005). Medicinal uses of ginger are also diverse and include the treatment of dyspepsia, colic, diarrhea, colds and flu, and poor appetite. Ginger is also recommended as an anti-inflammatory agent in rheumatic and muscular disorders and to increase longevity. Clinical trials support the use of ginger preparations to prevent motion sickness and vomiting in pregnancy, whereas the evidence is more ambiguous in the case of musculoskeletal disorders (Lee *et.al.*, 2007; K. C. Zancan *et.al.*, 2002).

Ginger products, such as essential oils and oleoresin, are commercialized for use in food and pharmaceutical processing. It is a common food additive in a number of foods and beverages and it is valued due to the volatile components especially the aromatic compounds which give a spicy, pungent and pleasant smell (Purnomo, 2010) and that volatile compounds responsible for the characteristics of ginger flavor (K. C. Zancan *et.al.*, 2002). The characteristic aroma of ginger is due to a volatile oil that is present in 1-3% quantities. Its pungency is attributed to ginger oleoresin (Lee *et.al.*, 2007).

2.2 Essential Oil

Essential oils are volatile and liquid aroma compounds from natural sources, usually plants. It is also highly concentrated essences of aromatic plants (Khairu Aizam, 2006). Essential oils are not oils in a strict sense, but often share with oils a poor solubility in water. The plants extracts are assumed to be more acceptable and for sure they are less hazardous compare to the synthetic compound (Jenny Jobling). Essential oils normally contain a complex mixture of organic compounds and they are largely composed of a range of saturated or partly unsaturated cyclic and linear molecules of relatively low molecular mass, and within this range a variety of hydrocarbons and oxygenated compounds occur (Ozel and Kaymaz, 2004).

2.2.1 Usage of Essential Oil

The essential oil has been used in many applications since long time ago. It has been found to be applied traditionally in medications, fragrances, flavours, preservatives and insect repellents. Essential oils often have an odor and are therefore used in food flavoring and perfumery. They are actually made up of many different volatile compounds and the makeup of the oils quite often varies between species. The oils are usually extracted by distillation, cold pressing, or extraction method.

These oils are being used widely as flavouring and masking agents in many foods, cosmetics and pharmaceuticals, in addition to aromatherapy (Mushtaq Ahmad and Salim-ur-Rehman, 2006). They are used in perfumes, cosmetics, soap and other products for flavoring food and drink, and for scenting incense and household cleaning products. So it means essential oils that are registered food grade materials, could be used as alternative anti-fungal and anti-bacterial treatments for fresh produce.

2.3 Methods of Extraction

2.3.1 Extraction of Ginger Oil by Soxhlet Distillation

Soxhlet extraction is one of the laboratory apparatus which is specially designed for the extraction of a lipid from the solid material and this method is commonly used (Ayuso *et.al.*, 1998) and has reached official status in many countries. Somehow, this Soxhlet Extractor is not limited to the extraction of the lipids only but this method can also be applied to any solution in which the desired compound has a given solubility in the solvent and the impurities are insoluble in the solvent. When a compound of low solubility needs to be extracted from a solid mixture a Soxhlet extraction can be carried out. The technique places a specialised piece of glassware in-between a flask and a condenser. The refluxing solvent repeatedly washes the solid extracting the desired compound into the flask.

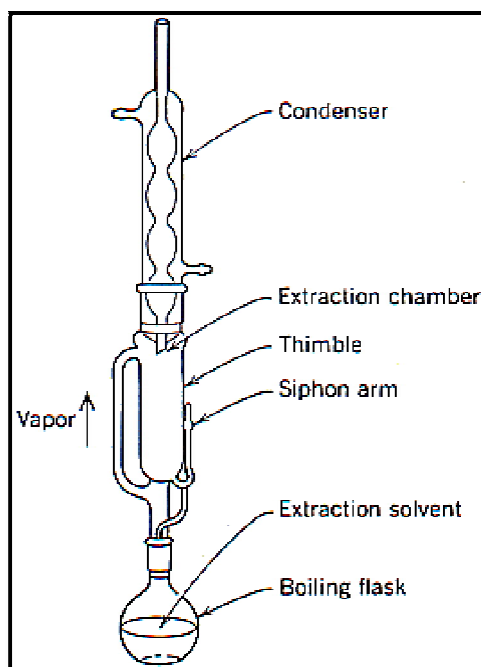


Figure 2.4: Soxhlet Extractor (adapted from technologylodging.com)

2.3.1.1 Theory of Soxhlet Extraction

Soxhlet extraction is actually one of the methods to extract essential oils such as *Origanum Onite* and *Coriandrum sativum L* (Ozel and Kaymaz, 2004). This extraction method use chemical solvents to extract oils by repeated washing or percolation with an organic solvents. The extraction of the ginger essential oils began when steam contact to the ginger in the extraction tank. The steam carried out the essential oils from the ginger out of the rhizome and go through the condenser. Then, the steam with the essential oils will be condensed into liquid phase and will be collected in the beaker. Soxhlet extraction shows more significant matrix compare to the ultrasonic method and that matrix effects depend heavily on the solvent used for the extraction (Schmeck and Wenclawiak, 2005). The advantage of this method is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled. Using stronger extraction conditions then the complete extraction is achieved (Schmeck and Wenclawiak, 2005). However, the Soxhlet extraction is about time consuming and labour intensive (Ozel and Kaymaz, 2004) where it is need large amount of solvents about up to 150 ml and large samples size which is up to 10 g (Saifuddin and Chua, 2003).

The most protruding advantages in using this Soxhlet method is when the sample phase is repeatedly bring into contact with fresh portion of the solvent, so then enhancing the displacement of the analyte from the matrix and no filtration is required (Ayuso *et.al.*, 1998).

Nevertheless, the Soxhlet extraction is still the preferred method because of its comparative extraction results despite the nature of matrix sample. Besides, Soxhlet extraction yields similar results with methods such as the supercritical fluid extraction (SFE), microwave-assisted extraction (MAE), accelerated solvent extraction (ASE) and ultrasonic methods. Some more the results also show small variations with low relative standard deviations (Lau *et.al.*, 2010).

However, the soxhlet extraction is still gave higher yield for certain components or materials to be extracted. It is proven in the Figure 2.4 when comparative studies were carried out, it was found that the highest efficiency of Petroleum Ether Extracts (PEE) from tobacco was obtained by Soxhlet extraction and this method is especially suited to sterols that cannot usually be extracted by Accelerated Solvent Extraction (ASE) or are seldom extracted by ultrasonically assisted extraction (Shen and Shao, 2005).

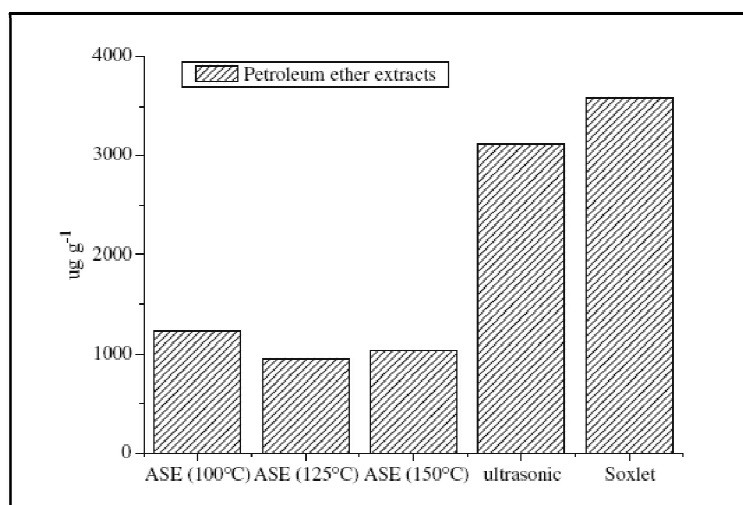


Figure 2.5 : Total PEE yields for ASE, Soxhlet Extraction and ultrasonically assisted extraction (Shen and Shao, 2005).

2.3.2 Ultrasound Assisted Extraction (UAE)

UAE can be used for extraction method with liquid solvents applied to analytes in solid matrices. This extraction method uses high-frequency sound to disrupt the target compound from the plant materials. Lately, there have been several reports on the application of ultrasonic methods in the extraction of various phytochemicals and determination of phenolic compounds (Gao and Chun-Zhao, 2005).

2.3.3 Microwave Assisted Extraction (MAE)

MAE is also one of the extraction methods which can reduce the extraction time and only consumes less volume of solvents and less sample amount (Gao and Chun-Zao, 2005; Saifuddin and Chua, 2003).

The major disadvantages of this MAE method however is that the solvent needs to be physically removed from the sample matrix upon completion of the extraction prior to further analysis. In some cases whereby samples are pretreated with activated copper bars to assist the extraction process because the removal of this copper is necessary for a cleaner extract. Although a subsequent purification step can be implemented to rectify this problem, there may be possibilities of losing analytes or inducing contaminants with additional cooling time for this extra handling. Furthermore, the sample allowance for analysis is limited to 1.0 g which is insufficient for a homogenous analysis (Lau *et.al.*, 2010)